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EPITAXIAL GROWTH OF SEMI-INSULATING GaAs ,

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Epitaxial Growth/Semi-insulating GaAs

October 1, 1977 to December 31, 1977

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OBJECTIVE

The objective of this program is to develop techniques for the vapor phase growth of high resistivity epitaxial layers of gallium arsenide on semi-insulating gallium arsenide substrates. A capability to grow such layers of high quality material with minimum structural defects and good surface quality for use as "buffer" layers prior to the growth of active layers for such devices as FETs and TELDs will eliminate the device performance problems caused by poor and inconsistent substrate quality.

DESCRIPTION OF PROGRESS

A. Introduction

During this quarter high resistivity epitaxial layers of both undoped and chromium doped GaAs have been grown on chromium doped semi-insulating (SI) GaAs substrates. A number of techniques are being investigated in order to develop a capability for characterizing both the substrate materials and the epitaxial layers.

B. Epitaxial Growth

The initial portion of this reporting period was occupied with the correction of a number of equipment failures, and the gradual elimination of the final traces of contaminants from the gas control system in order to reach a condition of stable operation. It was then possible to optimize the various operating parameters of the system such as gas flows, furnace temperatures, substrate location, etc. Following this initial phase, it was found that it is possible to grow high resistivity epitaxial layers without the addition of dopants to the gas stream. A number of samples of high resistivity material with and without the addition of chromium have been grown. A number of wafers having n and n⁺ layers grown on top of thick buffer layers have been produced and passed on for FET processing.

Undoped Epitaxial Layers: Under growth conditions which closely approximate growth conditions used in the older design reactor tube (See Status Rept. No. 1), it has proved possible to grow epitaxial layers of high resistivity at growth rates of approximately 25 microns per hour. Wafers 89, 94 & 95 are typical examples. These have epitaxial layers approximately 12 microns thick.

Attempts to characterize these layers by the usual Van der Pauw methods have been made. Resistivities of the order of $4-5 \times 10^4$ ohm-cm have been

measured. Attempts to determine carrier concentration from these experiments have failed due to insufficient equipment sensitivity. However, with substrate to epi-layer thickness ratios of approximately ten to twenty-to-one, measurements are influenced by the presence of the substrate.

A rough method of assessing the resistivity and breakdown voltage of the epi-layers which has been adopted is the following. Two tungsten wire probes are pressed onto the surface of the epi-layer spaced approximately 0.1 inches apart, and a voltage is applied between them using a curve tracer. This gives an I/V curve and shows the voltage breakdown of the material, if any. Good buffer layers will withstand up to 1500 V which is the maximum capability of the instrument. Fig. 1 shows traces produced by wafers A96, A95. The slope of the trace is indicative of the material resistivity.

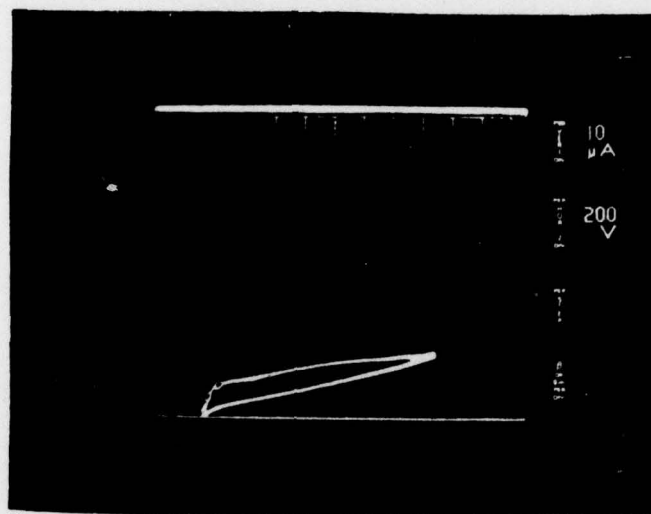
Chromium Doping: The original method selected to attempt chromium doping was to bubble a carrier gas (helium) through liquid chromyl chloride. Owing to a suspected leak in this part of the system, we are currently using a 500 ppm mixture of chromyl chloride (CrO_2Cl_2) in helium prepared in cylinders by AIRCO PRECISION GAS CO.

Several chromium doping runs have been made. Typical curve tracer breakdown photographs are shown in Fig. 2. (Wafers 89, 92, & 96). Attempts to measure the chromium content of these epitaxial layers by SIMS has been unsuccessful. We have sent several thick epi-layers for evaluation by spark source mass spectroscopy (SSMS). We will attempt using a weak spark and moving the wafer under the spark to limit erosion to less than 5 μm . Hopefully this will prevent the substrate from influencing the measurement.

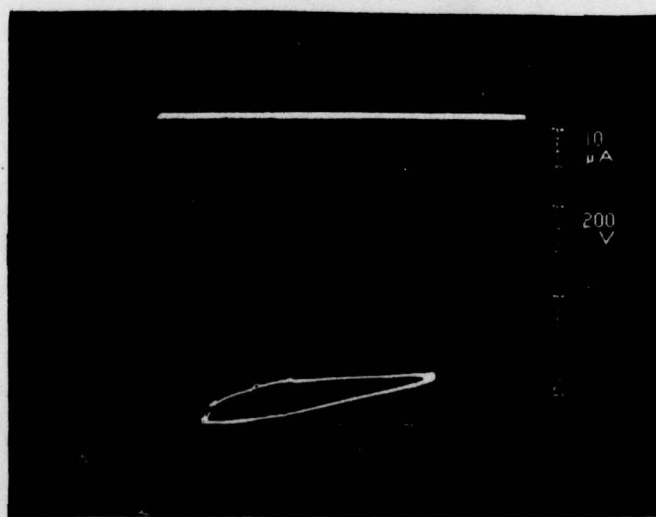
In Fig. 2, note that a dopant flow of 500 ccpm of CrO_2Cl_2 (500 ppm) produces a higher resistivity than a flow of 275 ccpm. Increasing the flow to 1000 ccpm resulted in the layer reverting to n-type with mid- 10^{14}-cm^{-3} carrier concentration.

The surface morphology of Cr-doped epi-layers grown in the modified reactor is excellent. This is in contrast to the layers grown in our older reactor.

Device Wafers: As the object of growing semi-insulating layers on SI GaAs substrates is to provide a superior surface for the growth of device wafers, a number of wafers suitable for GaAs FET fabrication have been grown. These have either a 12 μm thick undoped high resistivity layer or a chrome doped layer, followed by the n & n⁺ layers needed for FET fabrication.

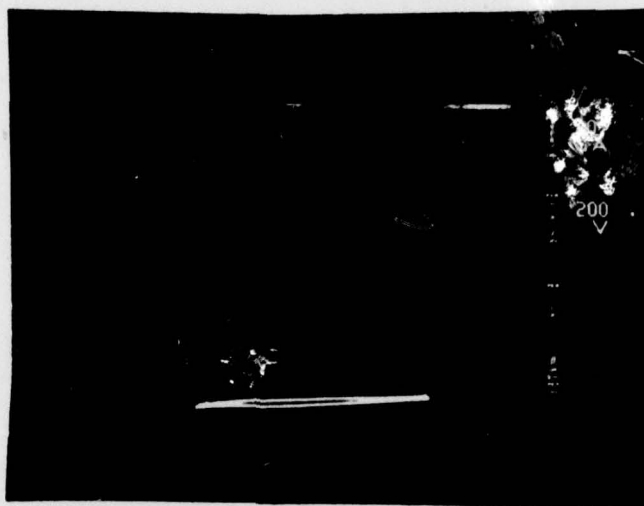


A94

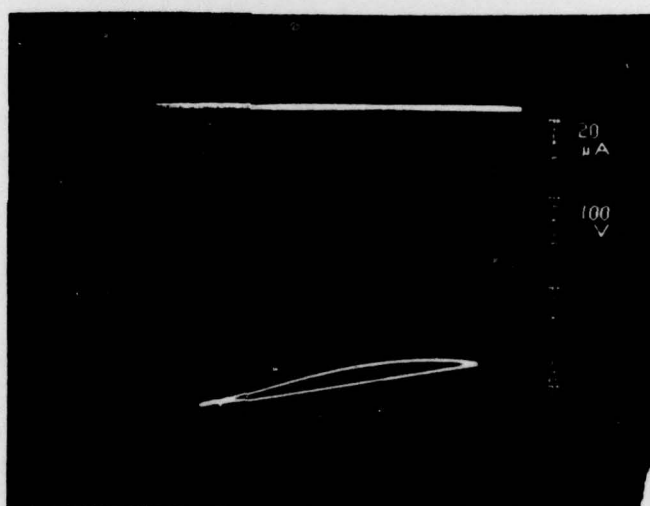


A95

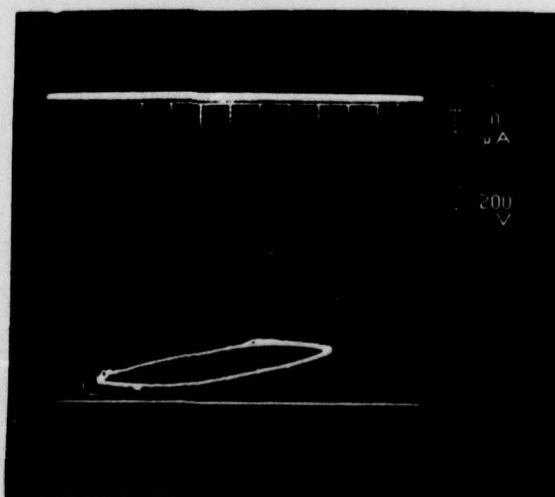
Fig. 1 2-point probe characteristic of undoped layers



A89
Dopant flow 500 ccpm



A92
Dopant flow 275 ccpm



A96
Dopant flow 500 ccpm

Fig. 2 2-point probe data for Cr-doped epi-layers.

These wafers are A63, A67, A72, & A73 with undoped and A69 with a chrome doped buffer layer.

Fig. 3 shows the carrier concentration profile of wafer A69.

C. Characterization of SI GaAs

Detection and identification of deep levels in SI GaAs is an important step towards characterization of the material. Cr and O are the two most common elements producing deep states although somewhat more insulating material has been obtained of late with Cr doping. Even at saturation, the Cr concentration in GaAs remains at the parts-per-million level making analytical detection of the element difficult. To date we have found that only spark source mass spectrometry (SSMS) has reliably detected Cr in GaAs.

However, it has been known for some time [1,2,3] that transitions to and/or from Cr acceptors in SI GaAs could be excited optically with a photon energy of about 0.9 eV. It is not clear in the literature whether holes or electrons are responsible for the increased photoconduction with this sub-gap illumination although this could be determined with a photo-Hall experiment. Nevertheless, a peak in the photoconductive spectral response at 0.9 eV would appear to be the "signature" of Cr presence in the lattice. The results of preliminary, room temperature measurements to test the use of photoconductivity for detection of Cr in currently-used substrate material are shown in Figure 4. Here the conductivity per μW of incident radiation (normalized photoconductivity) for three types of SI substrates is shown as a function of the photon energy.

The Sumitomo oxygen doped substrate served as a control in the experiment. The PC response of this material is somewhat featureless at 0.9 eV as expected. The Morgan substrate did show evidence for a peak at 0.9 eV. SSMS determined that the concentration of Cr in this sample was about 0.1 ppm ($2 \times 10^{15} \text{ cm}^{-3}$). The Laser Diode material exhibits a definite peak at 0.9 eV and was determined to have a Cr concentration of about 1.1 ppm ($2.3 \times 10^{16} \text{ cm}^{-3}$) by SSMS. In addition, the Laser Diode material showed lower dark conductivity than the Morgan and had the slow photoresponse characteristic of a high concentration of deep traps.

1. D. R. Heath, P. R. Selway and C. C. Tooke
British Journal of Applied Physics 1 (SER 2), 29 (1968).
2. W. Plesiewicz, J. Phys. Chem. Solids 38, 1079 (1977).
3. H. Stocker, Journal of Applied Physics 48, 4583 (1977).

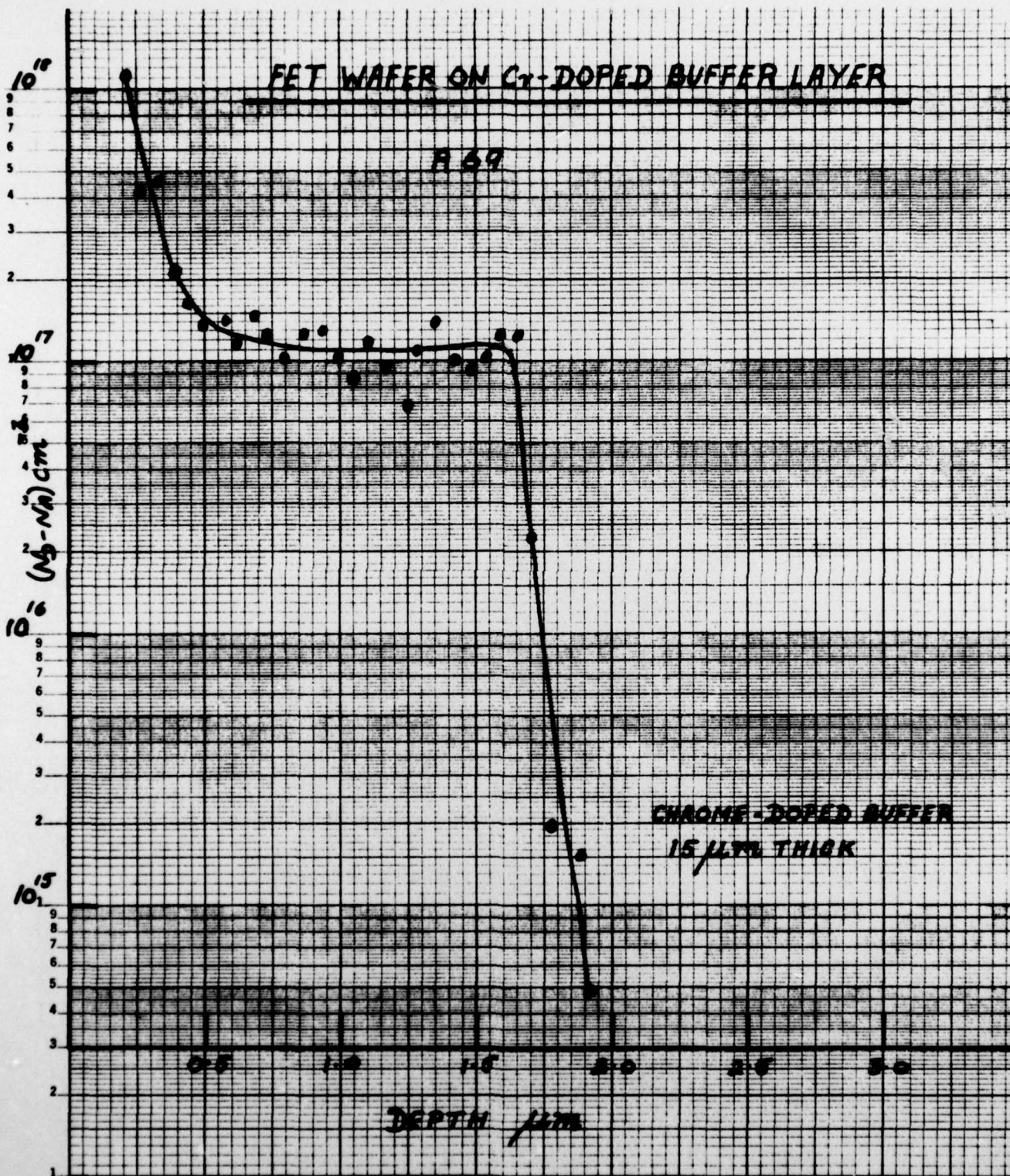


Fig. 3 Carrier concentration profile of wafer A69.

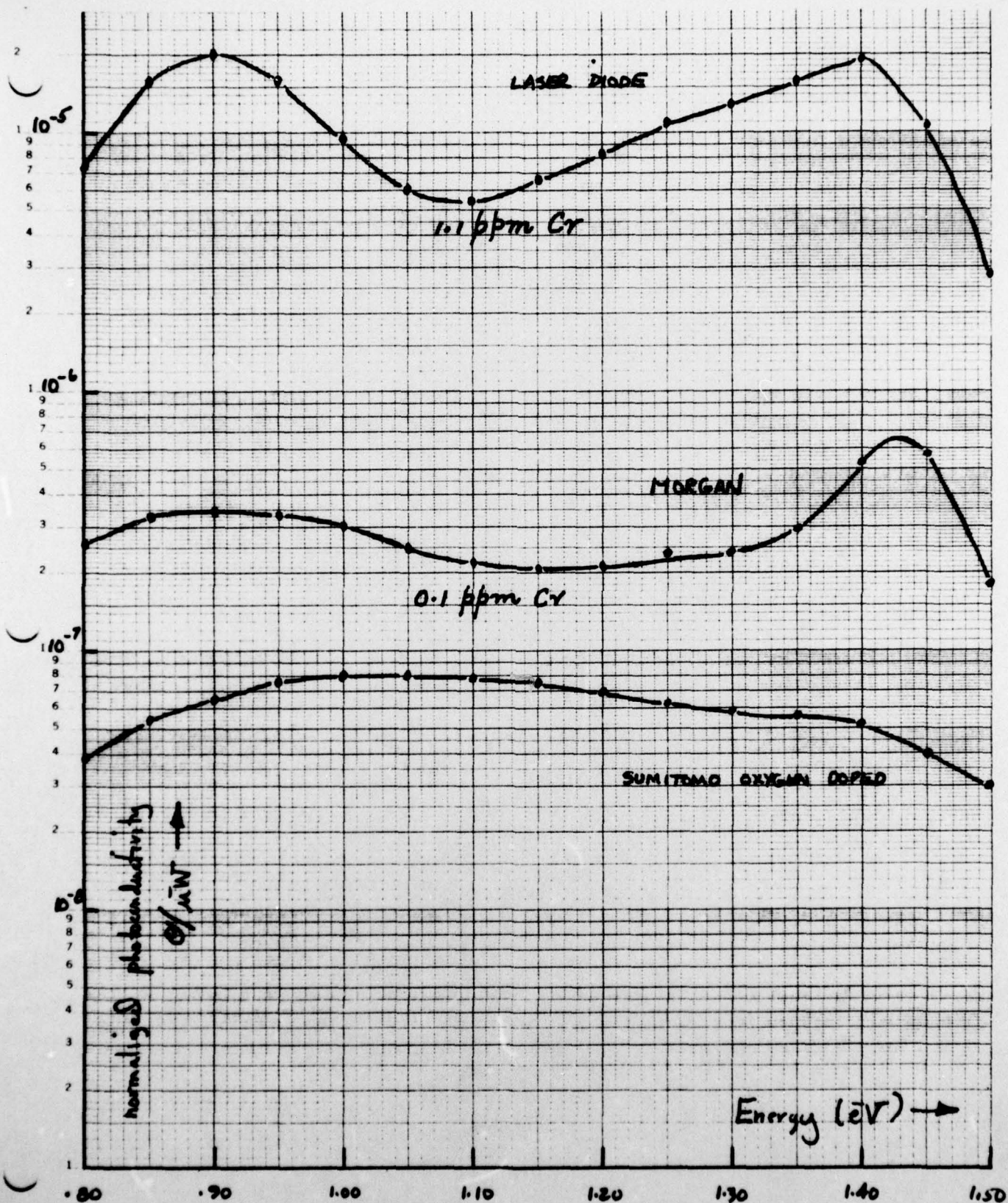


Fig. 4 Room temperature photoconductivity response of SI GaAs substrate. Note evolution of a peak at ~ 0.9 eV as Cr-content increases.

We conclude from this that photoconductivity measurements can be used to non-destructively detect the presence of Cr in GaAs substrate material. It is likely that other deep states could be resolved by extending the measurement to lower temperatures and photon energies.

Application of this method directly to the in-situ detection of Cr in thin epi-layers grown on SI GaAs substrates is not straightforward, however. Measurements of the optical absorption coefficient of Cr-doped material showed a negligible increase at 0.9 eV. Thus, the material remains optically transparent and any increase in photoconductivity could come from the Cr-doped substrate as well as the buffer layer. In view of the relative thickness, it seems unlikely that the effects of the buffer layer at 0.9 eV would be seen at all.

On the other hand, band edge illumination is absorbed very rapidly with depth because of the large value of the absorption coefficient. Behavior of the PC spectral response for the substrates tested was always similar. Conduction increased near the band edge and then dropped off as the photon energy increased. (See Fig. 4). This is interpreted as being due to a large value of the surface recombination velocity S . As the generated hole-electron pairs are confined to the surface, the photoconductivity is seen to decrease. Figure 5 shows the results of the same experiment (PC spectral response) for two SI epi films on the Morgan substrate. The characteristic dip above the band edge seen for the substrates was not observed indicating a lower value of S in the epi material.

The usefulness of photoelectronic measurements with sub-gap and band-gap radiation in characterizing GaAs:Cr has become apparent. Future effort will be directed toward lowering measurement temperatures for better resolution of deep levels. Also, photoluminescence measurements with excitation greater than the energy gap will be attempted for the detection of Cr in the buffer layer alone.

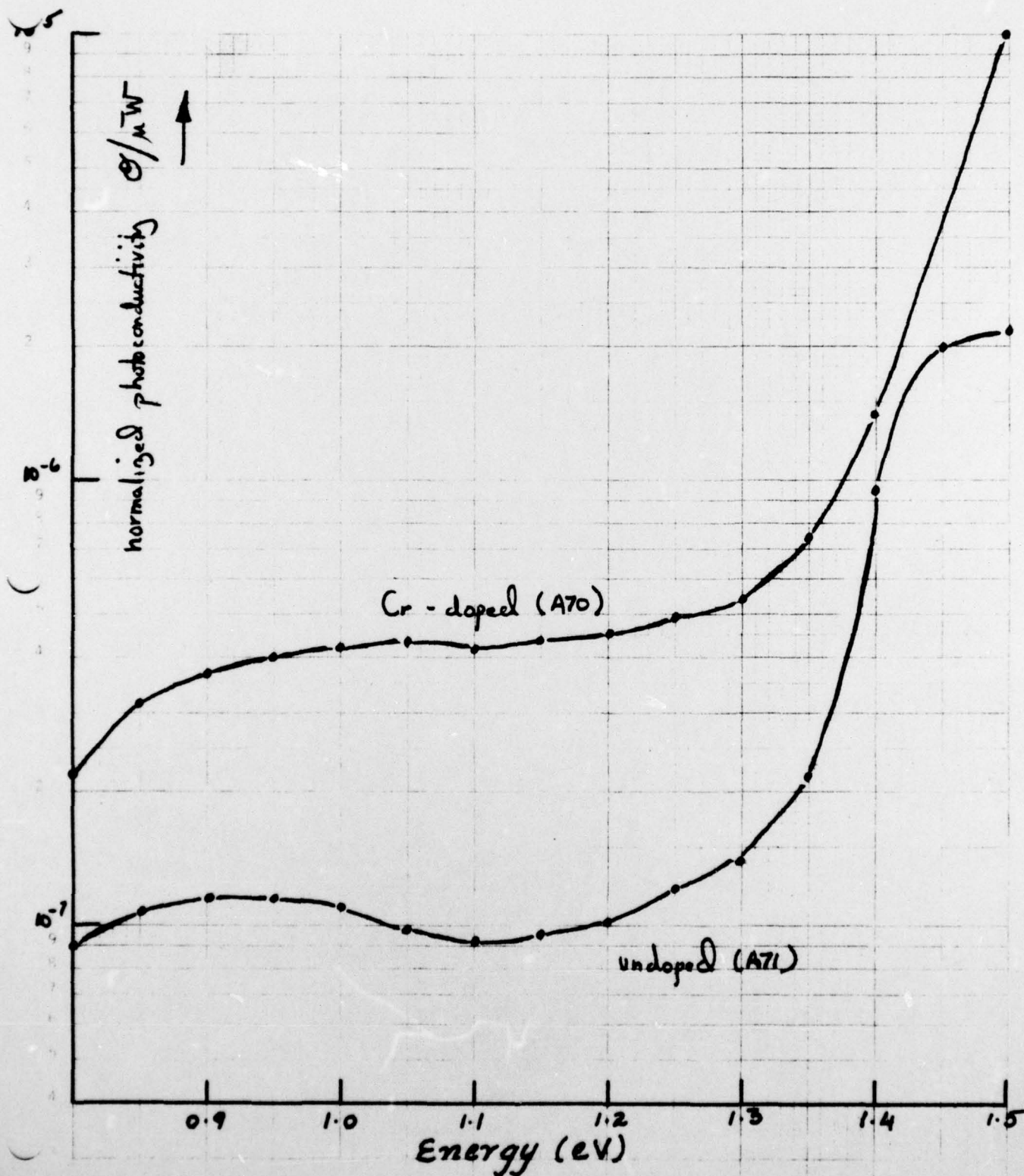


Fig. 5 Room temperature PC spectral response of two epi SI films on Morgan SI substrate.

D. Plans

1. Grow Cr-doped epi layers on a variety of SI GaAs substrates, including those showing conversion.
2. Study ion implantation into Cr-doped epi-layers. We will investigate implantation of Si and S atoms.
3. Continue photoconductivity studies.
4. Try photo-Hall measurements on both Cr-doped epi-layers and SI substrates.
5. Measure resistivity of Cr-doped epi-layers as function of temperature.
6. Grow thin n-layers on SI GaAs substrates with, and without epi buffer layers. Investigate traps at the n-layer interface to assess the efficacy of the buffer layer.
7. Grow TELD and FET wafers with Cr-doped epi buffer layers.
8. Investigate possibility of analysis of chrome doped layers using spark-emission spectroscopy.
9. Carry out further experiments to increase resistivity of buffer layers being produced.

CHANGES IN KEY PERSONNEL:

Dr. David Yaney started work on this program in October 1977. His responsibilities include characterization of SI GaAs and high resistivity epilayers.

SUMMARY OF SUBSTANTIVE INFORMATION DERIVED FROM SPECIAL EVENTS:

None during report period.

SPECIAL PROBLEMS ENCOUNTERED/ANTICIPATED:

None.

ACTION REQUIRED BY THE GOVERNMENT:

None.